

(12) UK Patent Application (19) GB (11) 2 275 625 (13) A

(43) Date of A Publication 07.09.1994

(21) Application No 9404045.8

(22) Date of Filing 03.03.1994

(30) Priority Data

(31) 93200623

(32) 05.03.1993

(33) EP

(71) Applicant(s)

Shell Internationale Research Maatschappij B.V.

(Incorporated in the Netherlands)

Carel van Bylandtlaan 30, NL-2596 HR, The Hague,
Netherlands

(72) Inventor(s)

Nigel Anthony Taylor

(74) Agent and/or Address for Service

D A H Bennett

Shell International Petroleum Company Limited,
Patents Licensing & Tr Mks Dvn, PO Box 662,
LONDON, SE1 7NE, United Kingdom

(51) INT CL⁵

B01D 53/04 53/14 , C10L 3/10

(52) UK CL (Edition M)

B1L LC LDJ L101 L102 L203 L219 L222 L318 L603
C5E EPG E181 E182 E192

(56) Documents Cited

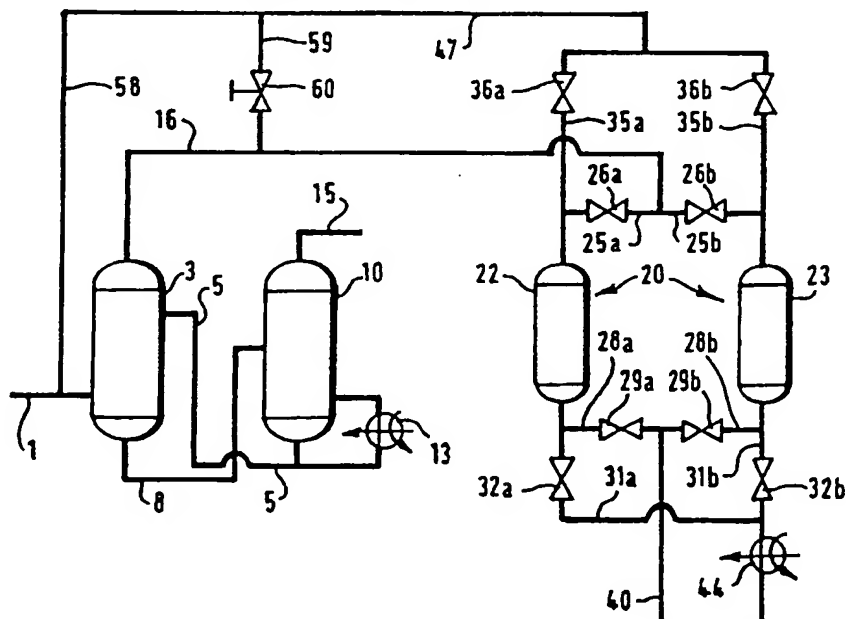
GB 0970567 A EP 0335034 A1 US 4259301 A

(58) Field of Search

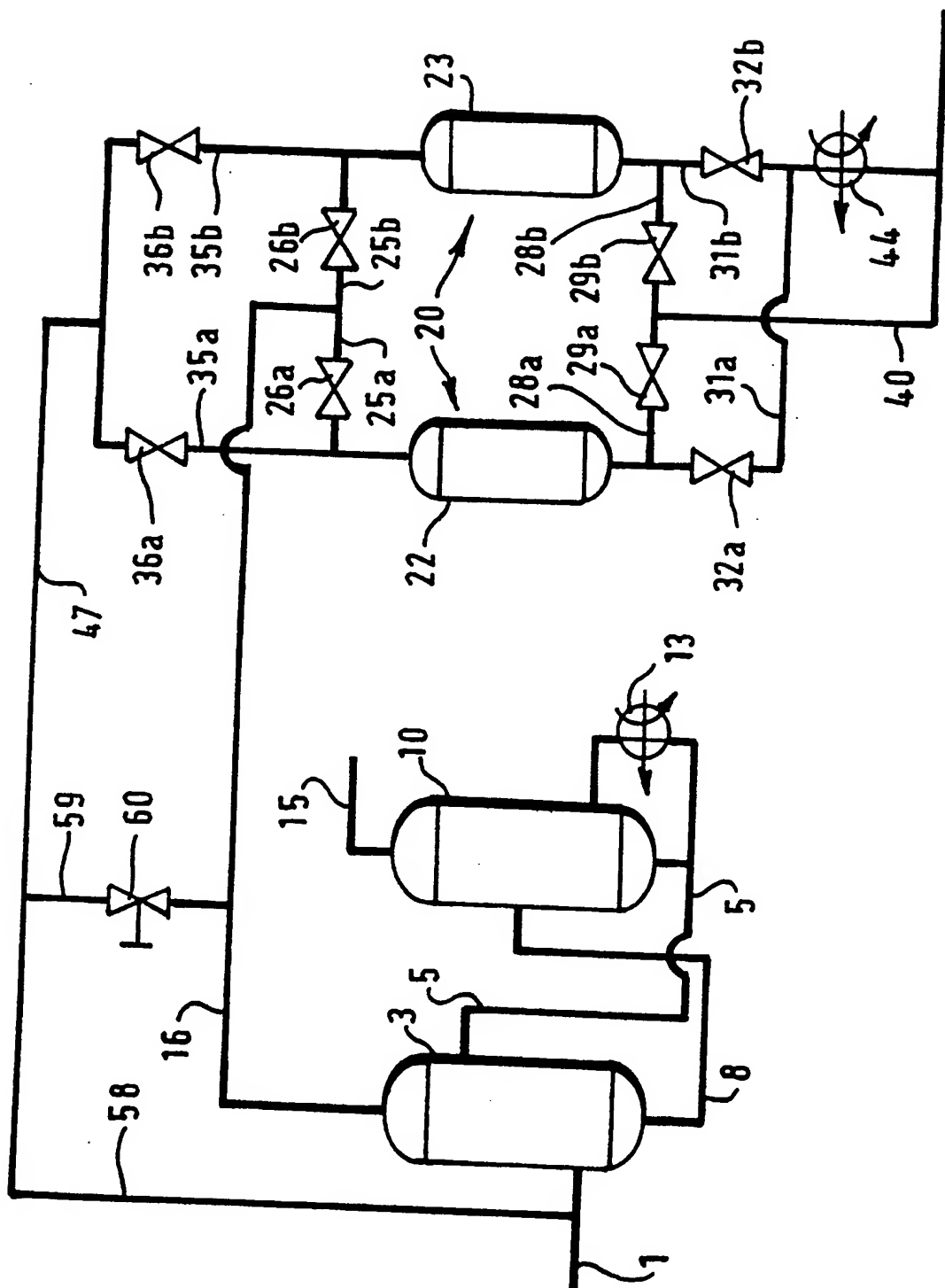
UK CL (Edition M) B1L LBA LBC LBD LC LDJ LDK LEA,
C5E EPG
INT CL⁵ B01D 53/00 53/02 53/04 53/14 53/34 , C10L
3/06 3/10
ONLINE DATABASES: WPI

(54) Removing hydrogen sulphide and organic sulphur compounds from a gaseous stream

(57) Method of removing hydrogen sulphide and organic sulphur compounds from a gaseous stream comprises contacting in an absorber (3) the gaseous feed stream with a regenerable aqueous absorbent (5) to obtain treated gas (16) and loaded absorbent (8) which is regenerated (10); contacting treated gas (16) with a solid adsorbent (22) to remove organic sulphur compounds from the treated gaseous stream and to obtain purified gas (40) and loaded adsorbent which is subsequently regenerated, wherein regenerating the solid adsorbent (22) comprises interrupting contacting the treated gaseous stream with the solid adsorbent (22), and contacting the solid adsorbent with heated purified gas to obtain regeneration off-gas (47) which is supplied to the gaseous feed stream upstream of the absorber (3).



GB 2 275 625 A



REMOVING HYDROGEN SULPHIDE AND ORGANIC SULPHUR COMPOUNDS
FROM A GASEOUS STREAM

The present invention relates to a method of removing hydrogen sulphide and organic sulphur compounds, such as carbonyl sulphide and mercaptans, from a gaseous stream comprising contacting in an absorber the gaseous stream with a regenerable aqueous absorbent to
5 obtain treated gas and loaded absorbent which is regenerated. It will be understood that when the gaseous stream further contains carbon dioxide, carbon dioxide will also be removed from the gaseous stream.

An example of such a gaseous stream containing hydrogen
10 sulphide and organic sulphur compounds, such as carbonyl sulphide and mercaptans, is natural gas. Typically natural gas contains up to 500 ppmv (parts per million by volume) of mercaptans. It will be understood that when the gaseous stream further contains carbon dioxide, carbon dioxide will also be removed from the gaseous
15 stream. A suitable regenerable aqueous absorbent is an aqueous solution of a chemical absorbent such as an alkanol amine, for example di-isopropanol amine or methyl diethanol amine, which contains a physical absorbent such as sulfolane.

It was found that such a method removes satisfactorily hydrogen
20 sulphide and that up to around 95 %vol of the organic sulphur compounds, and in particular mercaptans, is removed.

It is an object of the present invention to provide a method for even deeper removal of organic sulphur compounds.

To this end the method of removing hydrogen sulphide and
25 organic sulphur compounds from a gaseous stream according to the present invention comprises contacting in an absorber the gaseous stream with a regenerable aqueous absorbent to obtain treated gas and loaded absorbent which is regenerated; contacting treated gas with a solid adsorbent to remove organic sulphur compounds from the
30 treated gaseous stream and to obtain purified gas and loaded solid

adsorbent which is subsequently regenerated, wherein regenerating the solid adsorbent comprises interrupting contacting the treated gaseous stream with the solid adsorbent, and contacting the solid adsorbent with heated gas to obtain regeneration off-gas.

5 The solid adsorbent will provide removal 98 %vol or better of organic sulphur compounds in particular mercaptans. Suitable solid adsorbents are molecular sieves, and in particular zeolites of the types 5A and 13X.

10 The invention will now be described by way of example in more detail with reference to the accompanying drawing showing a line-up for the method of the invention.

15 Feed gas including hydrogen sulphide and mercaptans is supplied through conduit 1 to absorber 3, in which absorber 3 feed gas is contacted with lean regenerable aqueous absorbent in the form of an aqueous absorbent solution which includes sulfolane which is supplied to the absorber 3 through conduit 5.

20 Loaded absorbent having an increased content of hydrogen sulphide and mercaptans is removed from the absorber 3 through conduit 8 and it is supplied to a regenerator 10. In the regenerator 10 hydrogen sulphide and mercaptans are stripped from the loaded absorbent by means of steam produced in reboiler 13. A gaseous stream containing hydrogen sulphide and mercaptans is removed from the top part of the regenerator 10 through conduit 15 and passed on to a sulphur recovery plant (not shown). Lean absorbent is removed from the bottom of the regenerator 10 and passed through conduit 5 to the absorber 3.

25 Treated gas having a decreased content of hydrogen sulphide and mercaptans is removed from the absorber 3 through conduit 16. As the treated gas still contains a too large amount of mercaptans, 30 the mercaptans are removed by contacting the treated gas in an adsorbent system 20.

35 Adsorbent system 20 includes a first adsorber 22 and a second adsorber 23 each loaded with a suitable solid adsorbent, primary supply conduits 25a and 25b provided with valves 26a and 26b, primary outlet conduits 28a and 28b provided with valves 29a and

29b, secondary supply conduits 31a and 31b provided with valves 32a and 32b, and secondary outlet conduits 35a and 35b provided with valves 36a and 36b.

5 Treated gas is supplied through conduit 16 and the primary supply conduit 25a to the first adsorber 22, mercaptans are adsorbed on the solid adsorbent in the first adsorber 22, and purified gas leaves the first adsorber 22 through conduit 28a communicating with conduit 40. Valves 26b, 29b, 32a and 36a are closed.

10 When first adsorber 22 is used to remove mercaptans from the treated gas, the solid adsorbent in the second adsorber 23 is being regenerated. Regeneration is done by passing a bleed stream of the purified gas via heater 44 through conduit 31b to the second
15 adsorber 23. Regeneration off-gas leaves the second adsorber 23 through conduit 35b. The regeneration off-gas from the second adsorber 23 is passed via conduits 35b, 47 and 58 to the feed stream in conduit 1 upstream the absorber 3. Valves 32b and 36b are closed when the regeneration is completed.

20 When the solid adsorbent in the first adsorber 22 has been loaded with mercaptans valves 26a and 29a are closed, and valves 26b and 29b are opened to allow passing treated gas through the second adsorber 23. To regenerate the solid adsorbent in the first adsorber 22 valves 32a and 36a are opened. The regeneration
25 off-gas from the first adsorber 22 is passed via conduits 35a, 47 and 58 to the feed stream in conduit 1 upstream the absorber 3. Valves 32a and 36a are closed when regeneration of the solid adsorbent in the first adsorber 22 is completed.

The first adsorber 22 is used to remove mercaptans when the solid adsorbent in the second adsorber 23 has been loaded and is
30 regenerated, as described above.

The regeneration off-gas leaving the first adsorber 22 or the second adsorber 23 is passed to the feed stream in conduit 1 upstream the absorber 3. In this way the concentration of mercaptans in the feed is increased and an increased amount of

mercaptans is removed, so that build-up of mercaptans in the system is prevented.

If required a bleed stream of the regeneration off-gas can be passed through conduit 59 provided with valve 60 to the treated gas
5 in conduit 16.

In stead of using purified gas to regenerate solid adsorbent in the adsorbers 22 and 23, treated gas from conduit 16 can be used. To this end a bleed stream of treated gas is passed from conduit 16 to the heater 44 (conduit not shown) and from there through
10 conduit 31a to the first adsorber 22 or through conduit 31b to the second adsorber 23.

The solid adsorbent can in addition to removing mercaptans remove water from the treated gas. With the regeneration off-gas water is removed from the solid adsorbent, and to remove water from
15 the regeneration off-gas the regeneration off-gas is cooled in a cooler (not shown) and liquid water is separated in a gas-liquid separator (not shown) which cooler and separator are arranged in conduit 47.

C L A I M S

1. Method of removing hydrogen sulphide and organic sulphur compounds from a gaseous stream comprising contacting in an absorber the gaseous stream with a regenerable aqueous absorbent to obtain treated gas and loaded absorbent which is regenerated;
5 contacting treated gas with a solid adsorbent to remove organic sulphur compounds from the treated gaseous stream and to obtain purified gas and loaded solid adsorbent which is subsequently regenerated, wherein regenerating the solid adsorbent comprises interrupting contacting the treated gaseous stream with the solid
10 adsorbent, and contacting the solid adsorbent with heated gas to obtain regeneration off-gas.
2. Method according to claim 1, wherein the heated gas is heated purified gas.
3. Method according to claim 1 or 2, further comprising supplying
15 at least part of the regeneration off-gas to the gaseous stream upstream the absorber.

Patents Act 1977
Examiner's report to the Comptroller under Section 17
(The Search report)

Application number
GB 9404045-8

Relevant Technical Fields

- (i) UK Cl (Ed.M) B1L (LBA, LBC, LBD, LC, LDJ, LDK, LEA); L5E (EPG)
- (ii) Int Cl (Ed.5) B01D 53/00, 53/02, 53/04, 53/14, 53/34; C10L 3/06, 3/10

Search Examiner
MISS M M KELMAN

Date of completion of Search
29 MARCH 1994

Databases (see below)

- (i) UK Patent Office collections of GB, EP, WO and US patent specifications.

Documents considered relevant following a search in respect of Claims :-
1-3

- (ii) ONLINE DATABASES: WPI

Categories of documents

- | | |
|--|---|
| <p>X: Document indicating lack of novelty or of inventive step.</p> <p>Y: Document indicating lack of inventive step if combined with one or more other documents of the same category.</p> <p>A: Document indicating technological background and/or state of the art.</p> | <p>P: Document published on or after the declared priority date but before the filing date of the present application.</p> <p>E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.</p> <p>&: Member of the same patent family; corresponding document.</p> |
|--|---|

Category	Identity of document and relevant passages	Relevant to claim(s)
Y	GB 0970567 A (ENGINEERS & FABRICATORS INC) See page 2 lines 45-121	1, 2
Y	EP 0335034 A1 (UOP) See page 4 lines 12-17 and page 6 lines 37-56	1, 2
Y	US 4259301 A (EXXON) See whole document	1, 2

Databases: The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).